Reflection properties of pressed polytetrafluoroethylene powder

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The reflection properties of pressed polytetrafluoroethylene powder have been under investigation by the Radiometric Physics Division at the National Bureau of Standards for the past five years. This material has a great potential use, both as a standard of diffuse reflectance and as a coating for integrating spheres for applications in reflectance spectrophotometry and other signal-averaging devices. It possesses certain physical and optical properties that make it ideal for use in these applications. Techniques are given for preparing reflection standards and coating integrating spheres with the pressed powder. The effects of powder density and thickness on its reflectance are reported, and observations of possible problems with fluorescence that are due to the presence of contaminants in the powder are discussed. The absolute reflectance (6°/hemispherical reflectance factor relative to a perfect diffuser) is reported for the spectral range of 200–2500 nm. The directional/hemispherical reflectance factor relative to 6°/hemispherical reflectance is given for several wavelengths in the ultraviolet and visible spectrum and for angles of incidence between 5 and 75°. The bidirectional reflectance factor is reported for 300, 600, and 1500 nm at angles of incidence of −10, −30, −50, and −70° and at viewing angles at 10° intervals from −80 to +80°.

INTRODUCTION

The reflection properties of pressed polytetrafluoroethylene (PTFE) powder[1] in the ultraviolet, visible, and near-infrared spectral regions, combined with certain desirable physical characteristics, make this material exceptionally useful when there is a need for a good white diffuser or a standard of diffuse reflectance. The diffuse reflectance of pressed PTFE powder is remarkably high over the spectral range of 200–2500 nm and is probably higher than that of any other known material, its reflectance being 99% or higher over the spectral range of 350–1800 nm. It is particularly useful as a coating for integrating spheres commonly used in diffuse reflectance spectrophotometry. Historically, the most commonly used materials for this purpose have been magnesium oxide and barium sulfate powders or barium sulfate paint.[2] Much has been written on the optical properties of these materials, and the advantages and disadvantages of their use in spectrophotometric applications have been experienced by nearly everyone involved with reflectance spectrophotometry. Certainly, there is no longer any advantage in coating integrating spheres by the old method of burning magnesium and collecting the magnesium oxide smoke. The standards that still specify that reflectance measurements be reported on a photometric scale relative to smoked magnesium oxide should be rewritten to specify that reflectance measurements be reported on an absolute reflectance scale (relative to a perfect diffuser[3]). A review of publications on properties and reflection values of material reflection standards is given in Ref. 2.

The purpose of this paper is to make available the technical findings of several years of research on the optical properties of pressed PTFE powder. Other authors[4] have reported on the potential usefulness of this material as a reflection standard. The spectrophotometry group of the Radiometric Physics Division at the National Bureau of Standards (NBS) began studies of the reflection properties of pressed PTFE powder in 1975. At that time, this group was engaged in constructing a new reference spectrophotometer for diffuse reflectance[5] along with a number of accessory devices for measuring diffuse reflectance. These accessory devices include integrating spheres for measuring 6°/hemispherical reflectance factor, 45°/0° reflectance factor, and directional/hemispherical reflectance factor and a specular reflectometer.[6] At the same time, a more accurate absolute diffuse reflectance scale was being established by the auxiliary sphere or Van den Akker method.[7] A technique was developed for coating integrating spheres with the PTFE powder, and extensive measurements of the optical properties of this material were carried out over a five-year period. The results of these studies are reported here along with some descriptive details of the techniques used in preparing reflection standards from PTFE powder and its application to integrating spheres.

PREPARATION

The PTFE powder is somewhat lumpy as it comes in a shipping drum. It can be prepared for optical-coating purposes by reducing it to a uniform low-density powder. This can be accomplished with a blender or other chopping device, preferably one with stainless-steel blades and a glass container. The powder should be kept in glass containers and handled with tools made of materials such as stainless steel that are less likely to contaminate the material. The powder will adhere to itself on pressing, but it does not adhere well to metal, glass, or plastic. One technique for making it adhere to metal (or other materials) is first to coat the metal with a thin film of high-vacuum silicone grease. Once this is done, the powder can be pressed into place in thicknesses varying from 1 to 10 mm without much difficulty. The silicone grease has a low volatility and does not affect the reflection properties of the

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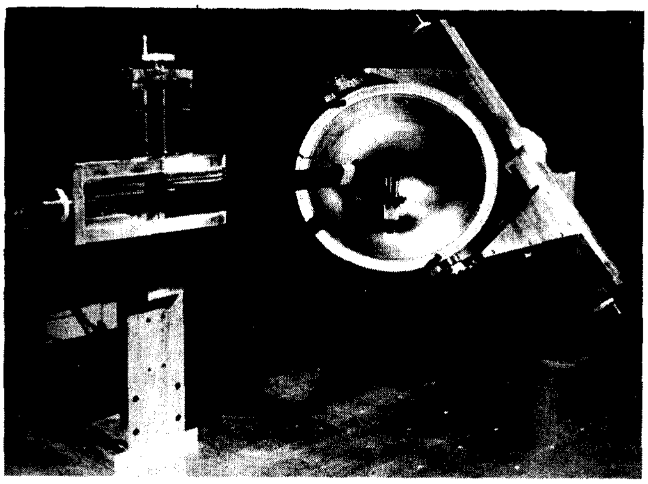

Fig. 1. The NBS integrating-sphere-coating apparatus showing the hemisphere manipulator with the electronically controlled tamping head used to press the PTFE powder into the hemisphere with a uniform radius.

PTFE powder because it contacts only a thin layer at the powder–metal interface.

PTFE powder has been successfully applied to integrating spheres ranging in diameter from a few centimeters to 45 cm and to large, flat surfaces used as reflection standards. The coatings are not affected by conditions of high humidity because the pressed powder repels water. Exposure to high-intensity ultraviolet radiation,[4] such as that encountered near a xenon arc, will slightly degrade the reflection properties of the coatings in the ultraviolet spectral region. The reflectance will be degraded as a result of contamination from smoke, dust, or other contaminants that may be present.

The time required for coating an integrating sphere of 20-cm diameter with a 6-mm-thick coating is typically about 4 h. This includes preparing the PTFE powder with a blender, trimming up the finished coating around the ports, and assembly of the usual two hemispheres. At NBS, spheres up to 45-cm diameter are coated with the aid of a special hemisphere manipulator (Fig. 1) that enables the technician to rotate and pivot the hemisphere about the center point of the sphere and pack the PTFE powder into the curvature of the hemisphere with a light pressure, using hand tools such as a stainless steel spoon or a round-bottom glass flask. In some special applications, such as in the preparation of an auxiliary sphere for determining the absolute reflectance of the sphere coating, the radius of curvature of the coating surface is carefully controlled by tamping the PTFE powder under an electronic tamping head before it is rolled to a final finish. A retainer ring is attached to the hemisphere flange during the coating procedure to retain the powder at the open edge of the hemisphere. When the coating is completed, this retaining ring is removed, and the two completed hemispheres are attached to each other by means of the flanges. The PTFE coating expands slightly at the hemisphere edge when the retaining ring is removed. This is an advantage because it results in the formation of a tight fit when the two hemispheres are combined to form the sphere.

As with any sphere-coating material, PTFE powder has its advantages and disadvantages. Some practice is required in order to master the coating techniques. The powder should be packed in lightly at first, to a depth of approximately 2–3 times the desired final thickness before it is pressed to a finish. The application of added powder to an already hard-pressed coating may result in a peeling and separation of the material into layers. The best results are obtained with a sand-blasted or ground-glass pressing tool, such as a round-bottom flask of small diameter. The coating should be done in a relatively clean, dust-free environment because the PTFE coating usually becomes electrostatically charged during the pressing and will hold small specks of dirt or lint. These can be picked up or removed with a small, clean artist's brush.

PTFE powder is very fine and easily mixes in the air about the working area. Although the powder is believed to be nontoxic, it is a good practice to use a dust mask to avoid unnecessary breathing of the material.

PTFE may form toxic gases at thermal decomposition temperatures above 400°C. These toxic products may be produced inadvertently by contact of the fine powder with a flame or other high-temperature source. For further information on the safe handling of fluorocarbon resins, refer to Ref. 8.

It should be noted that there are U.S. patents dealing with the use of fluorocarbons as coatings for integrating spheres. These U.S. patents are given in Ref. 9.

DENSITY

The reflectance of PTFE powder is influenced by the density to which the powder is pressed. This relationship is illustrated in Fig. 2, in which the results of a number of measurements of reflectance versus powder density are plotted. The reflectance scale in this illustration is a relative one with the data normalized at a density of 1.0 g/cm^3, because this is the density at which the powder reflectance is the highest. There seem to be no noticeable wavelength-related effects for wavelengths less than 2000 nm. The vertical bars in Fig. 2 show the spread in reflectance values at the 10 selected

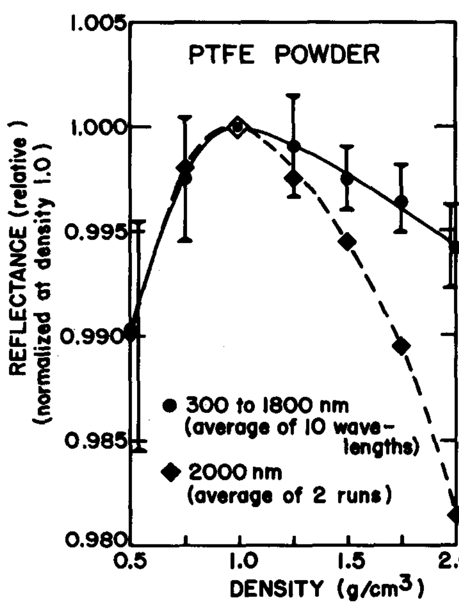

Fig. 2. Pressed PTFE powder reflectance as a function of powder density (10-mm thickness).

Table 1. Reflectance of Pressed PTFE Powder (0.8 g/cm^3) Relative to That of a 10-mm-Thick Layer at Each Wavelength

Thickness (mm)	Relative Reflectance		
	400 nm	550 nm	700 nm
1	0.991	0.989	0.986
2	0.995	0.994	0.993
4	0.998	0.998	0.997
6	1.000	1.000	0.999
8	1.000	1.000	1.000
10	1.000	1.000	1.000

wavelengths. The maximum reflectance is obtained by compressing the powder to a density of from 0.8 to 1.2 g/cm^3. The loose powder has a density of approximately 0.5 g/cm^3. A sample of the pressed powder having the maximum reflectance can easily be prepared by compressing 2–2.5 volumes of loose powder into 1 volume. This results in a pressed sample that is rather soft. A sample compressed to a density of 2 g/cm^3 will have a much harder surface and slightly lower reflectance. Higher densities require higher pressures, and a mechanical press must be used. As is shown in Fig. 2, the reflectance does not vary much with change in powder density for the density range of 0.8–1.2 g/cm^3. Repeated preparation of pressed samples having reflectances that are the same to within a few tenths of a percent can easily be achieved, and a fresh sample can be prepared in minutes. At wavelengths greater than 2000 nm the reflectance drops more rapidly with increasing powder density.

THICKNESS

The diffuse reflectance of pressed PTFE powder is influenced by the thickness of the powder layer. Its translucency is such that at least a 6-mm thickness at a density of approximately 1 g/cm^3 is required to obtain maximum reflectance. Table 1 shows the influence of thickness on the reflectance of PTFE powder at 400, 550, and 700 nm. These measurements were made with an aluminum backing having a reflectance of approximately 60%. Later measurements in the infrared out to 2500 nm indicate that 6- to 8-mm thicknesses and a density of 1 g/cm^3 are adequate in order to achieve maximum diffuse reflectance values for the pressed powder. Coatings this thick are not required in applications where the coating is used in a signal-averaging sphere. However, when the PTFE powder is used as a reflection standard, a 10-mm thickness will be certain to give the maximum reflectance at all wavelengths.

FLUORESCENCE

There has been no clear evidence to show that the PTFE powder itself fluoresces. However, coatings made from PTFE powder show a very weak fluorescence. This fluorescence may be associated with a slight contamination from the plastic bag used to contain the PTFE powder in its shipping drum. As a test of the contribution of fluorescence to the reflectance of PTFE powder, a specimen was first measured on a double-beam reflectance spectrophotometer with the use of monochromatic illumination of the PTFE powder and then with polychromatic illumination. In Table 2 the normalized difference D between the reflectances obtained by these two modes of illumination is given as $D = (R_m - R_p)/R_p$, where R_m is proportional to the reflectance of the PTFE powder sample relative to that of an aluminum mirror with monochromatic irradiation and R_p is the same relative reflectance with polychromatic irradiation. There appeared to be a slight amount of fluorescence with excitation at wavelengths less than 290 nm and emission in the wavelength range of 310–350 nm. Subsequent measurements with a spectrofluorometer[10] confirmed these conclusions.

The levels of fluorescence observed may be negligible for most radiometric and spectrophotometric applications. However, in applications in which a small amount of fluorescence can cause a significant error, it would be wise not to use any kind of pressed-powder coating without first checking for the presence of fluorescence that is due to contaminants, especially when ultraviolet radiation below 300 nm is involved.[11]

REFLECTION PROPERTIES

The 6°/hemispherical reflectance factor of a diffuse reflectance standard is probably the most important calibration. The term 6°/hemispherical reflectance refers to a measurement geometry in which the sample is illuminated by a collimated source incident upon the sample at 6° from the normal

Table 2. Data from Fluorescence Check

Wavelength (nm)	D	Wavelength (nm)	D
250	0.019	500	0.000
260	0.016	510	−0.002
270	0.022	520	0.000
280	0.018	530	0.000
290	0.005	540	0.002
300	−0.004	550	0.000
310	−0.018	560	0.000
320	−0.011	570	0.000
330	−0.021	580	0.005
340	−0.015	590	0.001
350	−0.009	600	0.006
360	−0.007	610	0.004
370	−0.006	620	0.001
380	−0.003	630	0.001
390	−0.002	640	0.000
400	−0.006	650	−0.002
410	−0.002	660	0.000
420	−0.002	670	−0.002
430	0.002	680	−0.002
440	0.002	690	−0.006
450	0.000	700	−0.003
460	0.002	710	−0.004
470	−0.002	720	−0.003
480	0.002	730	0.002
490	0.002	740	−0.008
		750	−0.007

Table 3. 6°/Hemispherical Reflectance Factor of a 10-mm-Thick Pressed PTFE Powder Relative to a Perfect Diffuser

λ (nm)	ρ	λ (nm)	ρ	λ (nm)	ρ
200	0.962[a]	950	0.994	2140	0.964
210	0.964[a]	1000	0.994	2150	0.965
220	0.967	1050	0.994	2160	0.967
225	0.968	1100	0.994	2170	0.970
230	0.969	1150	0.994	2180	0.973
240	0.971	1200	0.993	2190	0.975
250	0.973	1250	0.993	2200	0.977
260	0.976	1300	0.992	2210	0.977
270	0.978	1350	0.991	2220	0.978
275	0.979	1400	0.991	2230	0.978
280	0.980	1450	0.992	2240	0.977
290	0.982	1500	0.992	2250	0.977
300	0.984	1550	0.992	2260	0.976
310	0.985	1600	0.992	2270	0.976
320	0.987	1650	0.991	2280	0.975
325	0.988	1700	0.990	2290	0.974
330	0.988	1750	0.990	2300	0.972
340	0.989	1800	0.990	2310	0.971
350	0.990	1850	0.986	2320	0.970
360	0.990	1900	0.985	2330	0.968
370	0.991	1950	0.984	2340	0.966
375	0.991	2000	0.981	2350	0.965
380	0.991	2010	0.979	2360	0.964
390	0.992	2020	0.978	2370	0.963
400	0.993	2030	0.976	2380	0.963
450	0.993	2040	0.975	2390	0.962
500	0.994	2050	0.973	2400	0.962
550	0.994	2060	0.972	2450	0.961
600	0.994	2070	0.971	2500	0.960
650	0.994	2080	0.970		
700	0.994	2090	0.969		
750	0.994	2100	0.968		
800	0.994	2110	0.967		
850	0.994	2120	0.966		
900	0.994	2130	0.964		

[a] Extrapolated.

and in which the reflected flux is averaged by an integrating sphere-detector system. An angle of 6° off the normal is commonly used to allow for including the specular component in the measurement.

The 6°/hemispherical reflectance factor of samples of PTFE powder pressed to a thickness of 6 mm or more with a density of 0.8–1.2 g/cm^3 is very reproducible. For a set of samples made at different times from different lots of PTFE powder, a standard deviation of less than 0.001 in measured reflectance was found for the wavelength range of 400–750 nm. Further measurements will have to be made in order to establish what this deviation will be in the ultraviolet and infrared regions. The 6°/hemispherical reflectance factor of pressed PTFE powder is given in Table 3 for a PTFE coating 10 mm in thickness and having a density in the range of 0.8–1.2 g/cm^3. The data listed in Table 3 are plotted in Fig. 3. These data are reported on an absolute reflectance scale (relative to a perfect diffuser) and were obtained by the auxiliary sphere

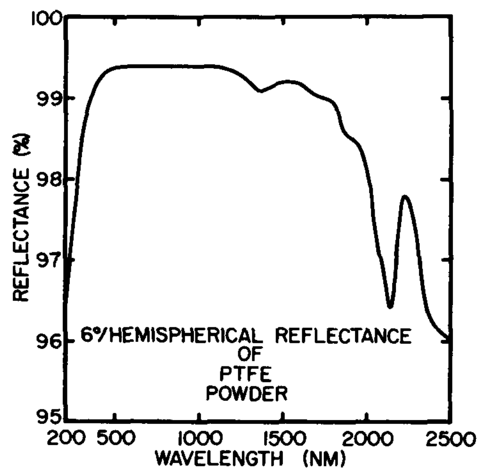

Fig. 3. The 6°/hemispherical reflectance factor (relative to a perfect diffuser) of 10-mm-thick, 1-g/cm^3-density pressed PTFE powder.

Table 4. Directional/Hemispherical Reflectance Factor of Pressed PTFE Powder for a 10-mm-Thick Coating (Relative to Hemispherical Reflectance at 6° Incidence)

Angle of Incidence	Reflectance					
	250 nm	300 nm	350 nm	450 nm	600 nm	750 nm
6°	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
10°	1.0003	1.0002	1.0001	1.0001	1.0001	1.0001
15°	1.0009	1.0005	1.0004	1.0002	1.0002	1.0002
20°	1.0017	1.0010	1.0007	1.0004	1.0004	1.0004
25°	1.0026	1.0015	1.0011	1.0007	1.0006	1.0006
30°	1.0038	1.0022	1.0015	1.0010	1.0008	1.0008
35°	1.0052	1.0030	1.0021	1.0013	1.0011	1.0011
40°	1.0066	1.0038	1.0026	1.0016	1.0014	1.0014
45°	1.0082	1.0047	1.0032	1.0020	1.0017	1.0017
50°	1.0099	1.0056	1.0038	1.0023	1.0020	1.0020
55°	1.0117	1.0065	1.0044	1.0026	1.0023	1.0023
60°	1.0134	1.0074	1.0049	1.0029	1.0025	1.0025
65°	1.0153	1.0083	1.0054	1.0031	1.0027	1.0027
70°	1.0170	1.0090	1.0058	1.0032	1.0028	1.0029
75°	1.0186	1.0097	1.0061	1.0032	1.0029	1.0029

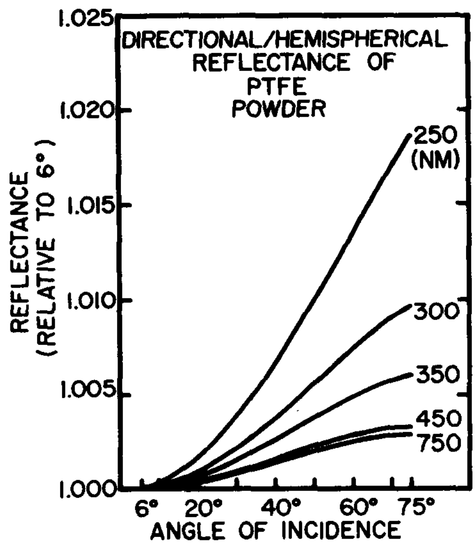

Fig. 4. The directional/hemispherical reflectance factor of pressed PTFE powder as a function of angle of incidence and wavelength.

method.[7] These absolute reflectance data are believed to be accurate to within ±0.002 on the basis of the analysis of the known sources of error as discussed in Ref. 7.

The directional/hemispherical reflectance factor of pressed PTFE powder given in Table 4 shows how the hemispherical reflectance of the material varies as a function of the angle of incidence. These measurements were made by means of an integrating sphere accessory to the NBS reference spectrophotometer for diffuse reflectance. This accessory consisted of a 45-cm-diameter sphere designed so that the sample can be mounted in the center of the sphere where it can be rotated to control the angle of incidence. The measurements were made at several wavelengths in the ultraviolet and visible spectra for both vertically and horizontally polarized sample beams. The results listed in Table 4 and illustrated in Fig. 4 are an average of the two polarizations. The values for either polarization did not vary by more than ±0.5% from the average. The most noticeable changes in reflectance as a function of angle of incidence occur in the ultraviolet spectral region. The data are relative to the 6°/hemispherical reflectance factor values.

The bidirectional reflectance factor of pressed PTFE powder listed in Table 5 shows how the reflectance varies over a wide range of viewing angles for four different angles of incidence. These measurements were made by means of the NBS specular reflectometer.[6] This instrument is ordinarily used for specular reflectance measurements as a function of angle of incidence. It can be used to measure the bidirectional reflectance of a diffuse sample in the plane of the incident sample beam. Measurements were made at 300, 600, and 1500 nm for angles of incidence of −10, −30, −50, and −70°. The angles of viewing were selected at 10° intervals from −80 to +80°. The bidirectional reflectance data reported in Table 5 are an average of the vertical and horizontal polarizations. The values for either polarization varied from this average by as little as ±0.2% to as much as ±20% depending on the combination of beam incidence and viewing angle. These data are relative to the reflectance values obtained at 0° (normal) viewing. The results listed in Table 5 are representative of the bidirectional reflectance of a "rough" surface prepared by pressing the PTFE powder with a coarsely ground glass plate. Samples pressed with a polished glass plate exhibit slightly higher values of reflectance, particularly at viewing angles greater than 75°. Figure 5 illustrates the bidirectional reflectance properties of pressed PTFE powder at 600 nm. Similar results are shown for barium sulfate powder in Fig. 6.

Further studies of the reflection properties of pressed PTFE powder are being made at NBS. Among these properties are the 45°/0° or 0°/45° reflectance factors for the visible spectral region. The results of these studies will be published at a future date.

Table 5. Directional/Directional Reflectance Factor (Bidirectional Reflectance) of 10-mm-Thick Pressed PTFE Powder

Angle of Viewing	Angle of Incidence 300 nm −10°	−30°	−50°	−70°	600 nm −10°	−30°	−50°	−70°	1500 nm −10° gnhr	−30°	−50°	−70°
−80°	0.763	0.792	0.872	1.148	0.743	0.761	0.827	1.048	0.702	0.830	0.820	1.045
−70°	0.836	0.867	0.937	–	0.822	0.840	0.898	–	0.760	0.882	0.855	–
−60°	0.882	0.914	0.980	1.074	0.872	0.891	0.946	1.019	0.819	0.918	0.886	1.000
−50°	0.919	0.949	–	1.022	0.909	0.931	–	0.987	0.875	0.952	–	0.992
−40°	0.946	0.979	1.000	0.996	0.939	0.967	0.982	0.974	0.920	0.974	0.936	0.980
−30°	0.967	–	0.993	0.982	0.963	–	0.980	0.971	0.959	–	0.945	0.980
−20°	0.988	1.005	0.991	0.981	0.986	0.998	0.984	0.974	0.978	0.980	0.966	0.985
−10°	–	1.000	0.994	0.988	–	0.998	0.991	0.983	–	0.990	0.982	0.995
0°	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000
10°	0.991	1.001	1.008	1.021	0.991	1.002	1.012	1.027	0.998	1.005	1.010	1.010
20°	0.982	1.001	1.022	1.052	0.983	1.003	1.027	1.067	0.986	1.012	1.018	1.060
30°	0.970	0.999	1.036	1.098	0.971	1.004	1.048	1.125	0.971	1.015	1.040	1.155
40°	0.960	0.997	1.056	1.163	0.957	1.004	1.076	1.212	0.942	1.018	1.066	1.270
50°	0.943	0.996	1.085	1.265	0.938	1.002	1.114	1.343	0.895	1.022	1.100	1.440
60°	0.921	0.992	1.125	1.426	0.914	1.000	1.168	1.549	0.832	1.030	1.155	1.695
70°	0.882	0.974	1.167	1.691	0.872	0.985	1.227	1.897	0.754	1.035	1.235	2.075
80°	0.817	0.937	1.214	2.190	0.802	0.946	1.291	2.551	0.660	1.040	1.340	3.090

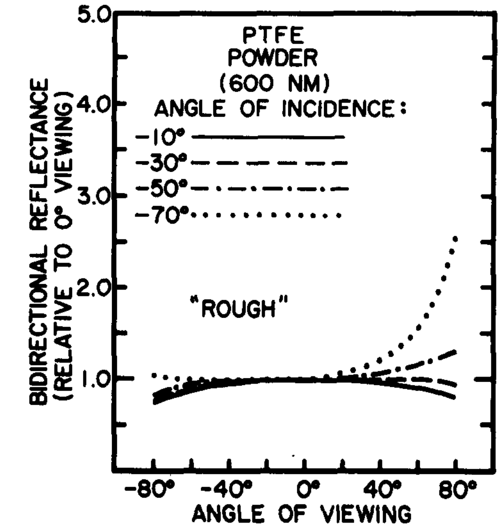

Fig. 5. The bidirectional reflectance factor at 600 nm of pressed PTFE powder having a rough surface finish.

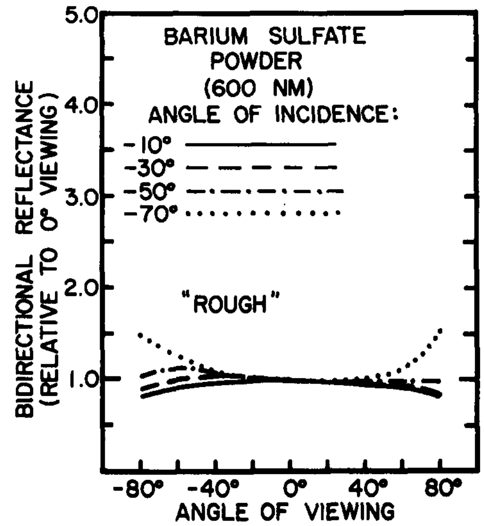

Fig. 6. The bidirectional reflectance factor at 600 nm of pressed barium sulfate powder having a rough surface finish.

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